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# Papers Accepted for Publication in Chromatographia

## Capillary Electrochromatography of Complex Plasma Matrix on a $C_{18}$/SCX Column using UV-Vis and Mass Spectrometric Detection

– by V. Spikmans, S. J. Lane, N. W. Smith

Separations of compounds in plasma were performed on a Hypersil Duet $C_{18}$/SCX capillary electrochromatography (CEC) column, utilizing an automated injection system combined with a short in-house designed and fabricated micro-electrospray CEC mass spectrometer interface. Protein precipitation was used prior to the CEC separations. More than two hundred separations of the corticosteroids Dexamethasone and Betamethasone 17-valerate, and Fluticasone Propionate in complex plasma matrix were performed on a single column under isocratic conditions. The method demonstrated good reproducibility, selectivity, sensitivity and high efficiencies. Linear calibration with good correlation was typical. Estimated detection limits in the low micromolar and nanomolar range for all compounds were obtained using UV-Vis absorbance (UV) and Electrospray Mass Spectrometric (ES/MS) detection respectively. Efficiencies for all compounds were typically 87,500 plates on a 25 cm column (350,000 plates $m^{-1}$) and increased with the number of plasma samples injected, up to 250,000 plates per column (1,000,000 plates $m^{-1}$). These very high observed plate counts may be artificially enhanced by the inadequate scan possibilities of the MS over very narrow chromatographic peaks.

## Enantioselective Liquid-Solid Extraction for Screening of Structurally Related Chiral Stationary Phases

– by E. Tobler, M. Lämmerhofer, W. R. Oberleitner, N. M. Maier, W. Lindner

An enantioselective liquid-solid batch extraction method is described for the screening of novel chiral stationary phases (CSPs) during optimization studies of chiral selectors derived form a common lead structure. Extraction enantioselectivity ($\alpha$) values can be calculated from the enantiomeric excess *ee*-values of the selectand, which are measured in the liquid phase by enantioselective HPLC. Extraction $\alpha$-values have been correlated with chromatographic $\alpha$-values. The influence was studied of several experimental parameters of the assay ($pH_a$, buffer concentration, temperature, selector/selectand and phase ratio) on the enantiomeric excess (*ee*) values of the selectands and the enantioselectivity of the CSPs, respectively. The derived statistically significant model has then been implemented to predict chromatographic $\alpha$-values of novel CSPs. For example, an *ee* of 89.3 % for DNB-Leu as selectand could be achieved in batch extraction for a novel synthesized but mechanistically similarly-acting CSP derived form quinine. This corresponds to a calculated extraction $\alpha$-value of 17.7. Based on this $\alpha_{extraction}$ a chromatographic $\alpha$-value of 28.8 was predicted by the linear correlation model; the experimental HPLC $\alpha$-value of 31.7 was in good agreement and demonstrated the validity of the proposed screening method. The method is particularly helpful in SO optimization studies.

## Development of Chromatographic Method for the Determination of Degree of Polymerisation of Polyglycerols and Polyglycerol Fatty Acid Esters

– by B. De Meulenaer, B. Vanhoutte, A. Huyghebaert

From polyglycerol samples, pure standards of tri and tetraglycerol were obtained using a column chromatographic technique. Polyglycerols were derivatised to tri-methyl silyl derivatives to obtain gas chromatographic identification of all linear, branched and cyclic diglycerol isomers and all linear and branched triglycerols. Moreover, quantitative gas chromatographic analysis of the di-, tri- and tetraglycerol content of a polyglycerol sample was developed.

Finally a method to characterise, on a quantitative basis, the polyglycerol content of polyglycerol fatty acid esters was developed. It consisted of saponifying the esters, without affecting the degree of polymerisation of the polyglycerols, followed by gas chromatographic analysis of the isolated polyglycerol fraction.

## Fast Solid Phase Extraction of Polychlorobiphenyls and Chlorinated Pesticide Residues from Mussels Using Sep-Pak Cartridges

– by M. V. Russo

A rapid method for the determination of chlorinated pesticides and polychlorinated biphenyls in mussels (Mytilus sp.) is reported. The mussel sample is homogenized and extracted with acetonitrile. The organic solution is concentrated and successively diluted with distilled water solution (12 g $L^{-1}$ NaCl). The organic compounds from water solution are adsorbed onto a $NH_2$ Sep-Pak cartridge.

The clean-up step, in which the polychlorobiphenyls and chlorinated pesticides are separated in different eluates, is achieved by passing 25 mL of a 40 % methanol aqueous solution through the $NH_2$ Sep-Pak and the $C_{18}$ Sep-Pak cartridges connected in series.

The polychlorobiphenyls are desorbed from the $NH_2$ Sep-Pak cartridge whilst the chlorinated pesticides are recovered from the $C_{18}$ Sep-Pak cartridge.

In the separation of polychlorobiphenyls from the chlorinated pesticides tested in this work, only aldrin, heptachlor and 4,4'-DDD are partially adsorbed with the polychlorobiphenyls onto the $NH_2$ Sep-Pak cartridge.

The average recovery is ≥ 95.0 % with a relative standard deviation ≤ 5.0 %. The limits of detection for different pesticides and polychlorobiphenyl congeners are 0.01 and 0.008 $\mu g\ kg^{-1}$. The final determination is carried out by capillary gas chromatography with ECD.